

# Annealing Dependences of Coercivity, Anisotropy Magnetic Field and Resistivity for Amorphous CoZrNb Films Deposited by DC Planar Magnetron Sputtering

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## ABSTRACT

Amorphous CoZrNb films have been deposited by the magnetron sputtering which can highly improve the utilization efficiency of magnetic alloy target with high permeability. The saturation magnetization  $4\pi M_s$  of the CoZrNb films was about 14 kG. The easy and hard directions for magnetizing the films were orthogonally arranged each other in the film plane. The coercivity  $H_c$  in the easy and hard directions decreased from 0.9 to 0.2 Oe with annealing in the rotating DC magnetic field  $H_A$ . However,  $H_c$  increased drastically to 20 Oe by heating at the annealing temperature  $T_A$  of 400 °C. With increasing  $T_A$  and  $H_A$ , the anisotropy magnetic field  $H_k$  gradually decreased from 12 to 1 Oe and the resistivity  $\rho$  also decreased from 200 to 150  $\mu\Omega$ -cm. Consequently, it was found that  $H_c$  and  $\rho$  depended strongly on  $T_A$  and  $H_k$  had definite relationships with both of  $T_A$  and  $H_A$ .

## INTRODUCTION

As the amorphous CoZrNb films have the excellent soft magnetic properties, their per-  
operation attracts much interest for high density magnetic recording heads. Recently, many  
works have been carried out on CoZrNb films deposited by sputtering technique and the depo-  
sition conditions to obtain excellent soft magnetism were studied.<sup>1-4</sup>

In magnetron sputtering, the CoZrNb films may be bombarded by high energy particles  
ejected from the target plane, and have also large uniaxial anisotropy induced by the direction  
of the magnetic flux in the sputtering apparatus during film deposition.

It is said that the uniaxial anisotropy of amorphous CoZrNb films is caused by pair ordering  
of the magnetic atoms<sup>5,6</sup> and can be improved significantly by annealing in a rotating magnetic  
field after film deposition.<sup>7,8</sup>

In this study, the relationship between the soft magnetism of the CoZrNb films and the  
annealing process has been systematically investigated in detail. The effects of the substrate  
bombardment with high energy particles on the soft magnetism of the films are also referred.

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## EXPERIMENTAL PROCEDURE

Figure 1 shows a schematic diagram of magnetron sputtering apparatus used in this study.  $H_M$ , magnetic field generated by a coil wound around the center pole of the magnetic yoke was 380 Oe and  $H_E$ , that generated by solenoid coil arranged outside of the cylindrical pyrex glass chamber was 100 Oe.  $H_E$  was applied to the plasma in order to increase the path length of the  $\gamma$ -electrons with cyclotron motion, which may be effective for ionizing the Ar gas in the vicinity of the target plane.<sup>9,10</sup>  $H_E$  is in the direction from the substrate to the target, while  $H_M$  is in the direction orthogonal to  $H_E$  and is parallel to the film plane, being useful for enlarging the eroded area of the target plane.

Figure 2 shows the magnetic flux distribution between the target and the substrate. As the directions of the magnetic fluxes near the target plane were perpendicular to the target plane,  $\gamma$ -electrons ejected from the target plane were confined uniformly and then target utilization efficiency was improved.<sup>10</sup>

The target was a 8at.%Nb-4at.%Zr-Co alloy disks of 99.9 % in purity, 120 mm in diameter and 3 mm in thickness.

After the chamber was evacuated to the background pressure of 0.1 mPa, argon gas of 99.999 % in purity was introduced into the chamber. Sputtering conditions are listed in Table 1. As the applied voltage  $V_A$  is lower than that of other magnetron sputtering at the same

Table 1 Sputtering conditions

applied voltage $V_A$ (V)	300
discharge current $I_D$ (A)	1
input power $P_i$ (W)	300
Ar pressure $P_{Ar}$ (Pa)	0.2
target-substrate distance $d$ (mm)	50
solenoid magnetic field $H_E$ (Oe)	100
substrate temperature $T_s$ (°C)	250
deposition rate $R_D$ (Å/min)	720

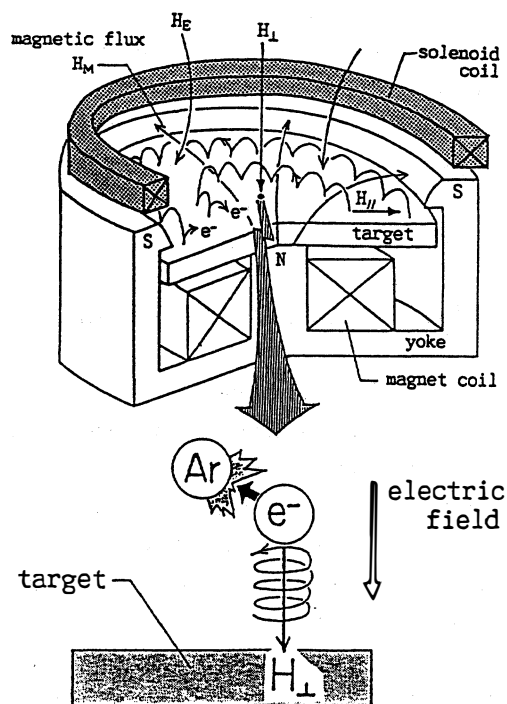


Fig. 1 Schematic diagram of the magnetron sputtering apparatus used in this study,

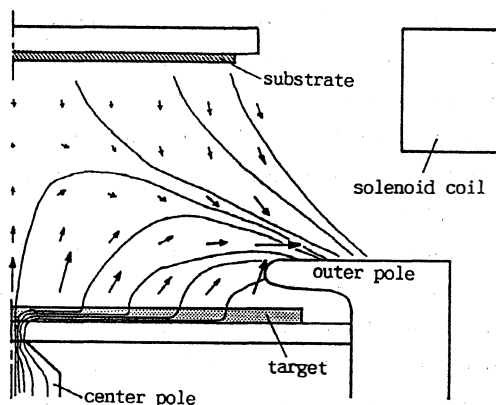


Fig. 2 Magnetic flux distribution between the target and the substrate.

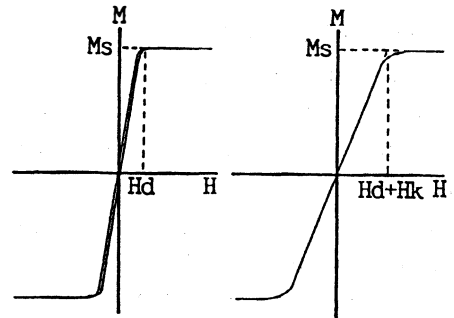
Ar pressure, it can be considered that the Ar atoms and ions with lower energy are reflected on the target plane and bombard to the surfaces of the substrate and the growing films. The specimen films with 1  $\mu\text{m}$  thickness were deposited on glass slide substrates.

After film deposition, annealing treatment was performed in a vacuum of 0.7 Pa in order to avoid oxidation of the films. The as-deposited films were heated at the annealing temperature  $T_A$  up to 400  $^{\circ}\text{C}$  in the rotating DC magnetic field  $H_A$  up to 500 Oe with an angular velocity of 30 rpm for an annealing time  $t_A$  up to 550 minutes.  $H_A$  is applied parallel to the film plane. The crystal structure was analyzed by X-ray diffractometry. The saturation magnetization  $4\pi M_s$ , the coercivity  $H_c$  and the anisotropy magnetic field  $H_k$  at room temperature were measured using a VSM and a M-H loop tracer, respectively.  $H_k$  was estimated from the M-H loops as shown in Fig. 3, where the difference between the fields necessary to saturate magnetically in the easy and hard directions was regarded as  $H_k$ . The resistivity  $\rho$  was measured with a four terminal method.

## RESULTS AND DISCUSSION

CoZrNb films deposited in this study have apparent uniaxial magnetic anisotropy and their easy direction of magnetization coincides with the direction of  $H_M$  in the magnetron sputtering apparatus. The easy and hard directions of the as-deposited films were orthogonally arranged each other in film plane. It has been well known that the magnetic anisotropy of the as-deposited films depended on the magnetic flux distribution on the substrate plane.<sup>7</sup> In the apparatus used in this study, since  $H_E$  perpendicular to the substrate plane was applied during deposition as shown in Fig. 2, the easy direction of the magnetization became perpendicular to the film plane. Therefore,  $H_c$  for easy direction and  $H_k$  may decrease by annealing in a rotating magnetic field. CoZrNb films have  $4\pi M_s$  of about 14 kG. Most of the as-deposited films were crystallographically amorphous.

Figure 4 shows the dependences of  $H_c$  and  $H_k$  on  $T_A$ .  $H_c$  in the easy direction of magnetization gradually decreased from 1 to 0.2 Oe with increasing  $T_A$  from 0 to 300  $^{\circ}\text{C}$ .  $H_c$  in the hard direction of magnetization was constant at 0.2 Oe even if  $T_A$  increased in the same range. However,  $H_c$  for both easy and hard directions



(a) easy direction (b) hard direction

Fig. 3 Estimation method for the anisotropy magnetic field  $H_k$ .

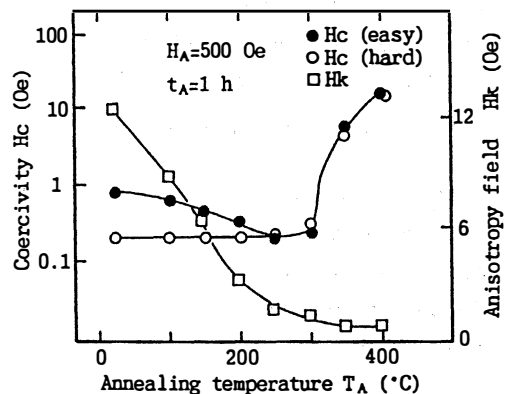


Fig. 4 Dependences of coercivity  $H_c$  and anisotropy magnetic field  $H_k$  on annealing temperature  $T_A$ .

increased apparently from 0.2 to 15 Oe with further increasing  $T_A$  from 300 to 400 °C. This may be due to the crystallization of CoZrNb films. However, the X-ray diffraction diagrams of films heated at  $T_A$  above 350 °C exhibited only a broad diffraction peak. On the other hand,  $H_k$  gradually decreased from 13 to 1 Oe with increasing  $T_A$  from 0 to 400 °C.

Figure 5 shows the dependences of the resistivity  $\rho$  on  $T_A$ .  $\rho$  was almost constant at 200  $\mu\Omega\cdot\text{cm}$  with increasing  $T_A$  from 0 to 300 °C. And then,  $\rho$  decreased apparently from 200 to 150  $\mu\Omega\cdot\text{cm}$  with increasing  $T_A$  from 300 to 400 °C. This may be due to the crystallization of CoZrNb films. This suggests that the microcrystallites which cannot be detected by the X-ray diffractometry may be included in the films. It was found that the crystallization temperature of the films was about 350 °C, being lower than that (530 °C) for the films deposited by FTS sputtering.<sup>3</sup>

Figure 6 shows the dependences of  $H_c$  and  $H_k$  on  $H_A$ .  $H_c$  in the easy direction of magnetization gradually decreased from 1 to 0.3 Oe with increasing  $H_A$  from 0 to 500 Oe.  $H_c$  in the hard direction of magnetization was constant at 0.3 Oe with increasing  $H_A$  in the same range. On the other hand,  $H_k$  gradually decreased from 12 to 1 Oe with increasing  $H_A$  from 0 to 500 Oe.

Figure 7 shows the dependences of  $H_c$  and  $H_k$  on the annealing time  $t_A$ .  $H_c$  in the easy and hard directions of magnetization were almost constant at 0.3 Oe with increasing  $t_A$  from 0 to 550 min.  $H_k$  decreased drastically from 10 to 1 Oe with increasing  $t_A$  from 0 to 500 min at  $t_A$  of 30 min. And then,  $H_k$  was almost constant at 1 Oe even if  $t_A$  increased from 30 to 550 min.

As seen in Figs. 4, 6 and 7,  $H_c$  in the easy direction and  $H_k$  decreased by annealing. This may be due to that the easy direction of magnetization changed from the direction perpendicular to the film plane to the direction parallel to the film plane and the internal stress caused by the

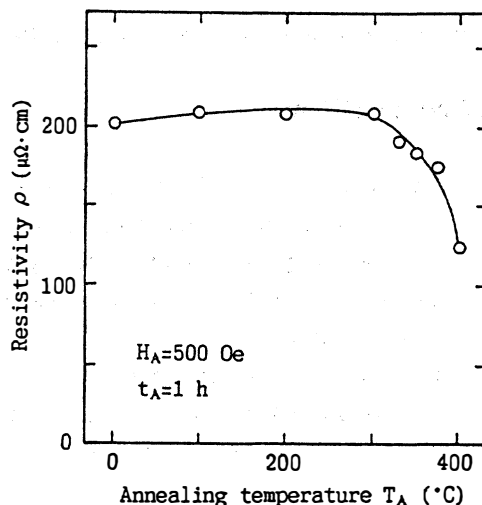


Fig. 5 Dependence of resistivity  $\rho$  on annealing temperature  $T_A$ .

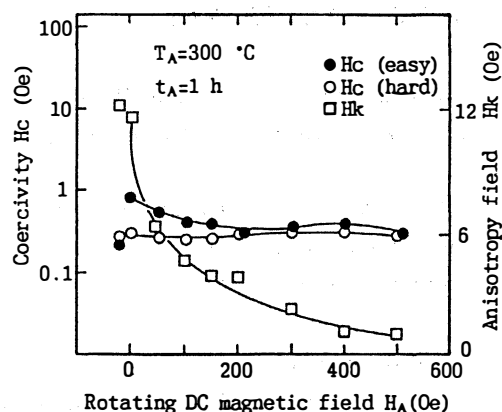


Fig. 6 Dependences of coercivity  $H_c$  and anisotropy magnetic field  $H_k$  on rotating magnetic field  $H_A$ .

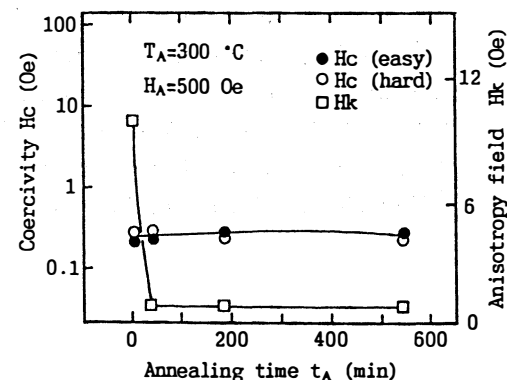


Fig. 7 Dependences of coercivity  $H_c$  and anisotropy magnetic field  $H_k$  on annealing time  $t_A$ .

bombardment of Ar ions and atoms reflected on the target surface was reduced.

## CONCLUSION

The dependence of the soft magnetism of amorphous CoZrNb films deposited by the magnetron sputtering on the annealing process has been investigated in detail.

The coercivity increased to 15 Oe by annealing at temperature above 350 °C. The resistivity measurement implied that this increase may be due to the crystallization of the amorphous grains in the films.

The anisotropy magnetic field decreased from 13 to 1 Oe with increasing the annealing temperature up to 400 °C in the rotating DC magnetic field up to 500 Oe. This may be attributed to the rotation of the easy direction of magnetization with regard to the film plane and the reduction of the internal stress in the films induced by the bombardment of Ar atoms and ions reflected on the target surface.

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## References

1. Y.Shimada and N.Saito, Jpn.J.Appl.Phys. **25**, 419 (1986).
2. H.J.de Wit, C.H.M.Witmer and F.W.A.Dirne, IEEE Trans. Magn. **MAG-23**, 2123 (1987).
3. M.Miura, H.Katahashi, K.Muramoto and M.Kajiyama, IEEE Trans. Magn. **24**, 2215 (1988).
4. K.K.Chon and J.H.Judy, J.Appl.Phys. **64 (10)**, 5495 (1988).
5. F.E.Luborsky and J.L.Walter, IEEE Trans. Magn. **MAG-13**, 953 (1977).
6. K.Y.Ho, J.Appl.Phys. **53**, 7831 (1982).
7. K.K.Chon, J.H.Judy; and J.M.Sivertsen, IEEE Trans. Magn. **MAG-23**, 2539 (1987).
8. Y.Yip, M.J.Vos, M.Lu, M.P.Dugas and J.H.Judy, IEEE Trans. Magn. **24**, 3072 (1988).
9. T.Takahashi, M.Yoneda and M.Naoe, Jpn.J.Appl.Phys. **28**, 379 (1989).
10. T.Takahashi, N.Ikeda and M.Naoe, IEEE Trans. Magn. **26**, 1494 (1990).